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# PATENT SPECIFICATION

NO DRAWINGS

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Date of Application and filing Complete Specification: Sept. 27, 1963.

No. 38210/63.

Application made in United States of America (No. 227566) on Oct. 1, 1962.

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Int. Cl.: —A 23 I 1/14

## COMPLETE SPECIFICATION

### Fatty Compositions

5 We, UNILEVER LIMITED, a Company registered under the laws of Great Britain, of Port Sunlight, in the County of Chester, England, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to aqueous

distribution and sale. Preferably, the composition should not require refrigeration, and for this reason dry topping mixes are advantageous. Spray-drying is one of the most economical means for obtaining such dry mixes in a free-flowing form. 50

Little is known about the requirements of a powder comprising fat, emulsifiers, and proteins with respect to the ideal physical

### ERRATA

#### SPECIFICATION No. 1,053,094

Page 2, line 86, for "2,117,983" read "2,177,983" 50

Page 2, line 86, for "2,117,984" read "2,177,984"

Page 5, line 33, for "monoglycerides" read "monodiglycerides" 15

THE PATENT OFFICE  
6th March 1967

25 to provide a powdered confectionery topping mix.

30 Such topping mixes have certain shortcomings. For example, they produce a relatively low overrun, e.g., less than 200%, or they lack the organoleptic properties of confectionery toppings based on whipping cream. Furthermore, the foams produced from these formulations are in some instances unstable on standing, i.e., the foam cells agglomerate to form large bubbles, resulting in an undesirable coarse, sponge-like structure. Additionally, 35 some products yield, on whipping, foams which tend to be sensitive to over whipping and break down or become curdy in texture if whipped excessively. 40

For commercial acceptability, a whipped confectionery topping composition must retain its desirable reconstituted properties over long periods of time under conditions of normal

70 by physical means such as constant agitation and the like. As a result, unless the process is carefully controlled, one obtains variability in the product, experiences difficulty in spray drying evenly and uniformly, or even may obtain an inferior product.

75 As an additional problem, dry confectionery topping mixes do not have indefinite shelf life. Even though they remain wholesome, they lose their ability to whip during storage. Little is known about the mechanism of this change. In some spray-dried toppings, loss of whippability during storage has been a severe problem. 80

85 The present invention provides compositions, preferably dry compositions, for use in preparing whipped confectionery toppings which go far to satisfy the various requirements indicated above.

The compositions of the invention comprise

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## COMPLETE SPECIFICATION

### Fatty Compositions

We, UNILEVER LIMITED, a Company registered under the laws of Great Britain, of Port Sunlight, in the County of Chester, England, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to aqueous or dry compositions for preparing whipped confectionery toppings.

It is known to prepare certain compositions which have properties similar to whipping cream after they are mixed with milk or water. These compositions can be pastes or free-flowing powders. The powders are preferred, since they are easy to handle and generally undergo less change during long-term storage. A typical composition in powder form has about 30—80% fat, 2—35% sugar, 5—30% non-fat milk solids and 3—20% of an emulsifier. Such compositions can generally be prepared by forming an aqueous emulsion and subsequently drying the emulsion to provide a powdered confectionery topping mix.

Such topping mixes have certain shortcomings. For example, they produce a relatively low overrun, e.g., less than 200%, or they lack the organoleptic properties of confectionery toppings based on whipping cream. Furthermore, the foams produced from these formulations are in some instances unstable on standing, i.e., the foam cells agglomerate to form large bubbles, resulting in an undesirable coarse, sponge-like structure. Additionally, some products yield, on whipping, foams which tend to be sensitive to over whipping and break down or become curdy in texture if whipped excessively.

For commercial acceptability, a whipped confectionery topping composition must retain its desirable reconstituted properties over long periods of time under conditions of normal

distribution and sale. Preferably, the composition should not require refrigeration, and for this reason dry topping mixes are advantageous. Spray-drying is one of the most economical means for obtaining such dry mixes in a free-flowing form.

Little is known about the requirements of a powder comprising fat, emulsifiers, and proteins with respect to the ideal physical state of these components for good whipping and for maintenance of whippability during storage of the powder. It is believed that the state of the emulsion prior to spray-drying, i.e., the fat globule size, the distribution of the protein, the distribution of the emulsifier, and other factors can influence the performance characteristics of the final dried product. The ready obtaining of an emulsion (prior to spray drying) which is stable and resistant to change while awaiting being fed to the spray drier is thus highly desirable. Known emulsions of this type, however, tend to separate readily into aqueous and fatty phases, and the emulsion must be maintained by physical means such as constant agitation and the like. As a result, unless the process is carefully controlled, one obtains variability in the product, experiences difficulty in spray drying evenly and uniformly, or even may obtain an inferior product.

As an additional problem, dry confectionery topping mixes do not have indefinite shelf life. Even though they remain wholesome, they lose their ability to whip during storage. Little is known about the mechanism of this change. In some spray-dried toppings, loss of whippability during storage has been a severe problem.

The present invention provides compositions, preferably dry compositions, for use in preparing whipped confectionery toppings which go far to satisfy the various requirements indicated above.

The compositions of the invention comprise

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a fat, a sweetening agent, a water dispersible protein, and a mixture of phosphoric acid esters of partial fatty acid glycerides with one or more additional emulsifying agents as hereinafter defined.

The said additional emulsifying agents are defined as emulsifying agents contained in one or more of the following classes:

- (a) saturated fatty acid monoesters of glycol
- (b) lactylated palmitic and/or stearic acid partial esters of glycerol
- (c) mixtures of unsaturated-fatty acid esters of polyhydric alcohols with lactylated saturated-fatty acid partial esters of polyhydric alcohols
- (d) mixtures of lactylated saturated- and unsaturated-fatty acid partial esters of polyhydric alcohols.

The invention includes, in addition to whipped or whippable compositions which are aqueous emulsions of the ingredients specified above, preferably of solids content about 35% by weight, also concentrates of such emulsions and dry powders obtained by drying them.

The various ingredients are combined in an aqueous emulsion, which may subsequently be spray-dried to provide a free-flowing powder having good handling characteristics. An excellent confectionery topping is obtained after reconstituting this powder with milk or water and subsequently whipping.

Glyceryl lactopalmitates or glyceryl lactostearates (about 4% to about 20% by weight of the complete composition) may be used in this invention in conjunction with from about 0.4% to about 2% by weight of a glyceryl lacto-oleate, a glyceryl monooleate, a glyceryl dioleate or mixtures thereof. It has been found in some instances that a composition which contains a combination of lactopalmitates or lactostearates with one of the unsaturated fatty acid esters is superior to a composition with only glycerol lactopalmitate therein. The glyceryl lactopalmitates, lactostearates or lacto-palmitate-stearates employed herein are formed by lactylating a mono- and diglyceride concentrate prepared from sources rich in palmitic and/or stearic acids. While the lactopalmitates are preferred, the lactostearates may also be satisfactory, and mixtures of the two can be used, as well as mixtures containing lower or higher fatty acid residues.

The esters of polyhydric alcohols and unsaturated fatty acids and the lactylated esters of polyhydric alcohols and unsaturated fatty acids which can be used in this invention include glyceryl lactooleate, glyceryl monooleate and glyceryl dioleate. These materials do not provide a satisfactory product if used as the major emulsifier, but markedly shorten whipping time, increase overrun and distinctly improve the whipped product texture if used

as a supplement to the lactopalmitate and/or lactostearate. A suitable glyceryl lactooleate can be prepared by lactylating a mono- and diglyceride concentrate made from oleic acid.

The phosphoric acid esters of mono- and diglycerides which are used in combination with the other emulsifiers can be formed by reacting phosphorus pentoxide separately with a partial glyceride having a saturated ester residue and with a partial glyceride having an unsaturated ester residue. However, these esters can also be provided from a single partial glyceride source. Other phosphorylating agents can be used instead of phosphorus pentoxide in making the phosphoric acid esters from the partial glycerides, for instance pyrophosphoric acid, metaphosphoric acid, ethyl metaphosphate, and phosphorus oxychloride. The preparation of phosphoric acid esters of partial glycerides is described in U.S. Patent Nos. 2,026,785, 2,117,983 and 2,117,984.

The partial glycerides used to prepare the phosphoric acid esters can be pure monoglycerides, a mixture of pure monoglycerides, or a mixture of mono- and diglycerides.

It is possible according to the invention to use a combination of two phosphoric acid esters—one of which the starting fatty acid is essentially saturated and the other in which the starting fat has an iodine value of 50 or higher. These two types of esters may be employed in a ratio of about 1:1, with the total ester concentration being 0.05% to 1%, preferably 0.1 to 0.5%. However, satisfactory results are also obtained if either type of phosphoric acid ester is employed alone.

The phosphoric acid ester with the saturated acid residue and the phosphoric acid ester with the unsaturated acid residue may be prepared separately. The results are also satisfactory if a partial glyceride with a saturated acid residue and a partial glyceride with an unsaturated acid residue are mixed together and a phosphoric acid ester thereafter prepared from this admixture. Accordingly, the iodine value of the mono- and diglycerides used to prepare the phosphoric acid esters can vary over a wide range, and good results are obtained from mono- and diglycerides derived from either completely hydrogenated fats and oils or liquid oils. An oil hardened to an IV of about 65 is preferred, since off-flavour problems may be encountered with extremely high IV oils. Extremely low IV products are not too satisfactory, since a high temperature must be maintained to keep the ester intermediates in a molten state.

The emulsifying system may consist of the phosphoric acid esters in combination with 4 to 20% by weight of the whole composition of a fatty acid mono-ester of a glycol. These esters are prepared by reacting any di-hydric alcohol with fatty acids, or fats containing fatty acids. One of the following specific

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procedures is generally employed: the methylation of fats and the subsequent reaction of the methyl esters with glycol; or the direct esterification of glycol and fatty acids. The

5   The aforementioned esterification usually forms, firstly, mono-esters, each containing one hydroxyl and one fatty acid group, and secondly, di-esters with both hydroxyls substituted with the fatty acid groups. The

10   The mono-esters are preferred over the di-esters; however, a mixture of mono- and di-esters is satisfactory. The fatty acid reactant preferably is saturated and has a chain length ranging from 12 to 22 carbon atoms, as, for

15   example, in lauric, myristic, palmitic, stearic, behenic and arachidic acids. Specific partial esters which are suitable for this invention are propylene glycol monostearate, propylene glycol mono-palmitate, propylene glycol mono-laurate, and propylene glycol mono-myristate, alone, or in admixture with the corresponding di-esters.

The aforementioned emulsifiers are advantageously used with 30% to 55% of fat, 25% to 45% of a sweetening agent, and 6% to 11% of protein.

A fat is incorporated into the confectionery topping mix to impart the desired creamy feel in the mouth. Fats for this invention should have a capillary melting point in the range of 35° to 40° C. Suitable fats are soyabean oil having an iodine value of about 80, partially hydrogenated cottonseed oil, coconut oil, and mixtures thereof.

35   The use of lecithin (1 to 3%) as an ingredient in the composition of this invention provides a product with improved texture. Suitable lecithin materials include natural soyabean lecithin, hydroxylated lecithins, and the ethanol insoluble fraction of natural lecithins. The latter product is preferred.

A sugar is generally the sweetening agent used in the present invention. Sucrose is the preferred compound. The amount of sweetening agent varies according to taste. Further, a portion of the sugar can be replaced by an artificial sweetening agent such as saccharine or a cyclamate, providing sufficient sugar remains to provide a spray-dried product

50   having an acceptable physical structure.

A caseinate, preferably sodium caseinate, is generally employed herein as the protein.

Emulsions prepared for spray-drying according to the present invention are quite stable, and maintain their physical state without appreciable change for prolonged periods of time, even longer than 4 hours. This facilitates greatly the spray-drying operation. Batches of emulsion can be made ahead of time, and

60   no special precautions need be taken to ensure that the beginning and end of an emulsion batch are alike in composition and physical state. One is assured of a uniform product from beginning to end of a batch without

resorting to costly equipment to ensure this result by physical means.

It is preferred, according to this invention, to maintain the pH of the composition within the range where optimum dispersion of the protein is obtained. For sodium caseinate, the optimum pH range is 6.5 to 7.5.

Aqueous compositions according to the invention, for whipping to produce a whipped confectionery topping should preferably have a solids content of about 35% by weight. The whipping characteristics are approximately the same whether the composition is prepared as a 60% solids concentrate and thereafter diluted to about 35% solids with cold milk before whipping, or whether it is spray-dried, diluted with cold milk to 35% solids and subsequently whipped. In order to reconstitute the dry compositions of the present invention, about 3.8 to about 4.2 fluid ounces (112—124 g.) of milk or water are added to about 2.2 to 2.7 ounces (62—77 g.) of spray-dried powder.

Thus, in accordance with the present invention, it is now possible to provide an improved whipped confectionery topping from a dry topping mix. Firstly, the manufacture of the mix, for example, the spray-drying operation, is simplified. Secondly, the dry powder, when mixed with liquid, whips rapidly, i.e., in less than about 3—4 minutes, to a stable foam with an overrun equal to or higher than whipped cream. Thirdly, the whipped confectionery topping has good structure; that is, it is firm and melts down properly in the mouth. Fourthly, the dry powder has improved stability of its whipping characteristics under the storage conditions generally encountered in distribution and warehousing.

Examples I, III and V below illustrate the invention while Examples II and IV show the disadvantageous effect of departing in different respects therefrom. Unless otherwise indicated, all parts and percentages in the specification are based upon weight.

#### EXAMPLE I

A confectionery topping mix was prepared from the ingredients indicated herebelow.

Ingredients	Parts	
Glyceryl lactopalmitate	7.0	
Glyceryl lactooleate	0.5	115
Groundnut oil, I.V. 65	41.7	
Phosphoric acid esters of cottonseed oil monodiglycerides (I.V. 65)	0.2	
Lecithin	1.0	120
Fat-soluble colour	0.3	
Sodium caseinate	8.0	
Vanilla flavour	0.037	
Sucrose	41.263	

A blend of 65.12 parts deodorized groundnut oil (I.V. 65), 32.55 parts glyceryl lactopalmitate, and 2.33 parts glyceryl lactooleate was prepared. This blend was subsequently

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5 treated for one hour at 50° to 55° C. with anhydrous sodium carbonate (22% by weight based on the oil-emulsifier blend). After vigorous agitation, the blend was filtered and deodorized for one hour at 150° C. under vacuum.

10 The aforementioned groundnut oil was prepared by hydrogenation with a nickel catalyst at 200° C. to I.V. 65 and by deodorization for four hours at 185° C. under vacuum. The aforementioned carbonate-treated blend and the remainder of the hydrogenated groundnut oil were admixed and the colour, phosphoric acid esters of monodiglycerides and lecithin were subsequently added at a temperature of 60 to 65° C. to provide a hot oil phase.

20 Sucrose and sodium caseinate were dry-blended and then dispersed in water at about 25° C. to form a 25% solids dispersion.

25 The hot oil phase was gradually added to the aqueous sucrose-caseinate in a stainless steel tank in conjunction with vigorous agitation. The resulting mixture was homogenized in a 2-stage homogeniser (1000 psig, 1st stage; 500 psig, 2nd stage) to provide an emulsion. This emulsion was subsequently spray-dried in a tower with the following conditions: emulsion feed rate, 0.28 lb/min.; inlet air temperature, 218.3° C.; exit air temperature 87.7—96.1° C.; and atomizing air pressure, 40 psig. A free-flowing powder was obtained with 0.8—1.3% moisture. This powder was passed through a 20-mesh sieve and subsequently dry-blended with the flavour.

35 The flavoured product was stored for 5 days at -1 to 4.4° C. and then stored at room temperature. After this storage, 67.5 g. of the powder were whipped with  $\frac{1}{2}$  cup of cold milk, using an electric mixer. The rate and extent of whipping were determined by measuring the overrun at different intervals. The results are as follows:

45	Time of Whipping (Min.)	Overrun (%)
	2	210
	3	255
	4	275

50 Some of the previously prepared powder was sealed in moisture-proof containers and stored for six months at room temperature. At the end of this time, the whipping procedure was repeated with the following results:

55	Time of Whipping (Min.)	Overrun (%)
	3	197
	5	234

60 It is manifest from this example that storage has only a minor effect upon the whipping properties of the powder prepared in accordance with the present invention.

#### EXAMPLE II

The same procedure, described in Example

I, was repeated except that the phosphoric acid esters were not incorporated into the confectionery topping mix. The emulsion separated into a fatty phase and an aqueous phase prior to spray-drying and it was necessary to agitate the emulsion after homogenization to maintain a suitable state for spray-drying. The powder therefrom, after storage at -1 to 4.4° C. for 5 days, was returned to room temperature and whipped with the cold milk. The following results were noted:

75	Time of Whipping (Min.)	Overrun (%)
	2	224
	3	255
	4	267

80 These results showed that the product may be prepared without the use of the phosphoric acid ester of mono-diglycerides, provided special processing precautions are observed, and that it whipped satisfactorily after its preparation and a five-day tempering period.

85 As in Example I, dry powder of this example was stored for six months. Its whipability after this storage period was measured as indicated herebelow.

90	Time of Whipping (Min.)	Overrun (%)
	3	150
	5	150

95 It is evident that the whipped confectionery topping powder was relatively unstable over the storage period when the phosphoric acid ester of mono-diglycerides was omitted.

Accordingly, Example I and II demonstrate that the use of a phosphoric acid ester of a mono- and diglyceride provides a superior end product. Without these phosphoric acid esters, the whipped topping powder has relatively poor stability under storage conditions. This is surprising in view of the large concentration of stabilizing agents which are employed. In contrast, the confectionery topping powder, with the phosphoric acid esters therein, has excellent stability.

#### EXAMPLE III

A composition was prepared with the following ingredients and spray-dried in the same way as described in Example I:

	Parts	
Cottonseed oil (I.V. 65)	49.79	
Propylene glycol monostearate	9.00	
Phosphoric acid esters of cottonseed oil mono-diglycerides (I.V. 65)	0.20	115
Hydroxylated lecithin	1.00	
Butylated hydroxyanisole	0.01	
Sodium caseinate	10.00	
Sucrose	30.00	120

No problems were encountered in either preparing or spray-drying the emulsion. The powder therefrom was stored for six days at 1.7° C. and then for two days at room

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temperature. Subsequent to this storage, 70 grams of powder were whipped with  $\frac{1}{2}$  cup of cold milk and vanilla flavour. The results are as follows:

5	Time of Whipping (Min.)	Overrun (%)
	2	190
	3	245
	4	265

10 Although the texture and flavour of the whipped material of this example were different from these of the product in Example I, the confectionery topping was quite acceptable.

#### EXAMPLE IV

15 A similar product was prepared as in Example III, except that additional sucrose was employed instead of the phosphoric acid esters of monodiglycerides. The emulsion prepared for spray drying was relatively unstable and the necessary emulsification was maintained by physical means prior to the emulsion being forced through the spray-drier nozzle. After the storage period at 1.7° C. and at room temperature, a whipped topping was provided from the powder and the following results were noted:

	Time of Whipping (Min.)	Overrun (%)
	2	147
	3	172
30	4	180

35 It can be seen from Example III and IV that the omission of phosphoric acid esters of monoglycerides created certain process problems. It also resulted, in this instance, in an inferior end product.

#### EXAMPLE V

40 A dry confectionery topping mix composition is prepared in the manner described in Example I, except that glyceryl monooleate is substituted for the glyceryl lactooleate. No difficulty is experienced with preparation of the emulsion prior to spray-drying. The spray-dried powdered confectionery topping mix obtained has excellent whipping characteristics as initially prepared and retains these

characteristics with little loss over a storage period of 6 months.

#### WHAT WE CLAIM IS:—

1. A composition for preparing a whipped confectionery topping said composition comprising a fat, a sweetening agent, a water dispersible protein, and a mixture of phosphoric acid esters of partial fatty acid glycerides with one or more additional emulsifying agents as hereinbefore defined. 50
2. A composition according to claim 1 which is in the form of dry powder. 55
3. A composition according to Claim 1 or 2, wherein the additional emulsifying agent comprises a saturated fatty acid mono-ester of a glycol. 60
4. A composition according to Claim 1 or 2, wherein the additional emulsifying agent comprises a mixture of a lactylated glycerol partial ester of a saturated fatty acid with an unsaturated fatty acid ester of a polyhydric alcohol. 65
5. A composition according to Claims 1 or 2, wherein the additional emulsifying agent comprises a mixture of a lactylated glycerol ester of a saturated fatty acid and a lactylated unsaturated fatty acid ester of a polyhydric alcohol. 70
6. A composition according to Claim 5, wherein the additional emulsifying agent comprises lactylated glyceryl oleate and a mixture of lactylated glycerol esters of palmitic and stearic acids. 75
7. A process for providing a composition according to claim 1 which comprises mixing phosphoric acid esters of partial fatty acid glycerides with a fat, a protein dispersed in water and an additional emulsifying agent as hereinbefore defined, followed by spray-drying the mixture. 80
8. A dry confectionery topping mix substantially as described in any of Examples I, III or V. 85

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